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PATENT SPECIFICATION

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(54) IMPROVEMENTS IN NICKEL-BASE ALLOYS

(71) We, GENERAL ELECTRIC COMPANY, a corporation organized and existing under the laws of the State of New York, United States of America, of 1 River Road, Schenectady 12305, State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates in general to castable high-temperature alloys and is more particularly concerned with a new nickel-base alloy having an unique combination of mechanical properties, stability characteristics and resistance to localized pitting and to ordinary hot corrosion in high-temperature corrosive environments. This invention is also concerned with a novel method by which articles of this alloy can be produced.

A nickel-base alloy which has been used quite successfully in aircraft engine applications is disclosed and claimed in U.S. Patent 3,615,376.

A nickel-aluminide type coating provides adequate protection from oxidation at high temperatures for buckets and other jet engine parts made of this alloy, but it has now been found that under substantially more severe environmental conditions resulting from use of alkali-metal-containing distillate or treated residual fuel, castings or articles of this alloy suffer catastrophic, localized corrosion or pitting. This mode of attack is unique among commercial nickel-base superalloys and is totally different from the hot corrosion attack which is characteristic of aircraft jet engine operation.

The merit of this special alloy compared

to other nickel-base alloys is such that if this tendency toward pitting could be effectively eliminated even at the cost of some loss of overall hot corrosion resistance, the resulting alloy would still be very attractive for certain gas turbine applications.

This invention in both its method and article aspects is predicated upon several discoveries we have made and a basically new concept rooted in those discoveries. Thus, we have found that the localized, catastrophic, hot-corrosion attack to which nickel-base alloys of this type are vulnerable is associated with the localized concentrations of molybdenum and tungsten in the MC carbide phase. When these carbides are present at the surface of the alloy casting article, pitting is initiated at those MC carbide sites. We have further found, however, that the amounts of molybdenum and tungsten are highly critical in producing this unusual corrosion effect and that the pitting tendency is effectively eliminated by reducing the combined total of molybdenum and tungsten in the carbide phase to less than about 15 percent. Additionally, we have found that such limitation of these two elements in the carbide phase can be readily accomplished without an offsetting disadvantage or penalty to any desirable characteristic of the alloy through the addition of tantalum, columbium or hafnium or mixtures thereof. If used in combination the amounts of these elements should aggregate in the range of 1.5 to 3.5%. If used individually, however, the amounts of these elements should be in the following ranges:

| | |
|-----------|-------------|
| Tantalum | 2.0 to 3.0% |
| Columbium | 1.0 to 1.5% |
| Hafnium | 2.0 to 2.5% |

These maxima and minima are governed by

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microstructural stability and carbide control considerations, respectively.

These findings have led to the new concept of displacing molybdenum and tungsten from the carbide phase to the alloy matrix to bring the total of these elements as carbides below the critical upper limit. In method or process terms, this concept is implemented through the new step of adding the requisite amount of the displacing element to the alloy suitably, but not necessarily, at the melt stage.

We have also discovered that the new alloys of the invention do not lose their good hot corrosion resistance when subjected to the usual heat treating operation in a vacuum. This is in sharp contrast to the alloys of this general type known in the prior art, where the depletion of chromium from the surface during vacuum heat treatment enhances hot corrosion attack. The time and temperature schedule is suitably designed in accordance with prior practice, but either vacuum or a neutral gas atmosphere can be used and it is not necessary or generally desirable to coat the workpiece in preparation for the heat treatment operation.

A cast, nickel-base, superalloy article of this invention can have a unique combination of mechanical properties, microstructural stability characteristics and resistance to localized pitting. In preferred form, this article also has resistance to general hot corrosion.

Accordingly the present invention provides a superalloy consisting of, by weight, 13.7 to 14.3 percent chromium, 9.0 to 10.0 percent cobalt, 4.8 to 5.5 percent titanium, 2.8 to 3.2 percent aluminium, 3.7 to 4.3 percent tungsten, 1.0 to 1.5 percent molybdenum, 0.01 to 0.02 percent boron, 0.02 to 0.10 percent zirconium, 0.08 to 0.20 percent, preferably 0.08 to 0.12 percent, carbon, and 1.5 to 3.5 percent of a mixture of tantalum, columbium and hafnium or, if individually, 2.5 to 3.0% Ta, 1.0 to 1.5% Cb and 2.0 to 2.5% Hf, balance nickel. The alloy in the form of a casting or article such as a gas turbine bucket consists of a matrix, an α' precipitate and a monocarbide (MC) phase, distributed through the matrix, which consists of titanium, molybdenum, tungsten and/or tantalum and/or columbium and/or hafnium in proportions such that the combined total of molybdenum and tungsten in the carbide constitutes less than 15 percent of the carbide phase.

According to the method suitable for this invention, the nickel-base superalloy article generally described above is made by first preparing an ingot of the composition in the amounts stated above. As a second step, the

ingot is remelted and cast to a form of the size and shape of the desired article. As the final step in the production of the preferred form of article of this invention, the article is heat treated in a vacuum or a neutral atmosphere, suitably in accordance with the appropriate time and temperature heat treatment schedule.

The present invention will be further described, by way of example only, with reference to the accompanying drawings in which:—

Figure 1 is a photomicrograph of a portion of a nickel-base superalloy specimen exhibiting the effect of localized hot corrosion attack (magnification 25 diameters);

Figure 2 is an X-ray scanning image photograph showing the concentration of tungsten at point (b) in the scale of the blister formed by the localized attack;

Figure 3 is a photograph like that of Fig. 2 showing the concentration of molybdenum at point (b);

Figure 4 is a photograph like that of Fig. 2 showing the concentration of tungsten at point (a) in the scale;

Figure 5 is another photograph like that of Fig. 2 showing the concentration of molybdenum at point (a);

Figure 6 is still another photograph like that of Fig. 2 revealing the absence of tungsten in the scale at point (c) in the normally attacked region of the specimen;

Figure 7 is another photograph like that of Fig. 2 revealing the absence of molybdenum in the scale at point (c);

Figure 8 is a Larson-Miller plot of the stress-rupture properties of a nickel-base superalloy René 80 and includes two alloys of the present invention;

Figure 9 is a Larson-Miller plot like that of Fig. 8 of two additional alloys of this invention; and,

Figure 10 is a third Larson-Miller plot of still other alloys of this invention.

Examples

The corrosion test results represented by Figures 1—7 were obtained in an experiment involving the investment casting of a five-pound ingot of René 80, a commercially available nickel-base superalloy disclosed and claimed in U.S. Patent 3,615,376, which has the following nominal composition:

| | |
|------------|-------------|
| Cobalt | 9.5 percent |
| Chromium | 14.0 " |
| Aluminium | 3.0 " |
| Titanium | 5.0 " |
| Molybdenum | 4.0 " |
| Tungsten | 4.0 " |
| Boron | 0.015 " |
| Carbon | 0.17 " |

Zirconium
Nickel

0.03 "
balance

with salt, retarding evaporation of the coating.

5 The ingot was sectioned and corrosion discs were subjected to heat treatment which consisted of heating for two hours at 2225°F (vacuum) and then for four hours at 2000°F (vacuum) and then for four more hours at 1925°F (vacuum) and finally for 16 hours at 1550°F in argon. Some of the corrosion discs were then coated with sodium sulfate (0.5 milligram per square centimeter) and hung with an unsalted disc in a pot furnace at 1700°F in air. The furnace liner, a closed-end mullite tube, contained a few grams of molten sodium sulfate and the temperature of the furnace and the position of the samples were adjusted so that the reservoir salt was at a temperature as high as or slightly higher than corrosion discs throughout the heating period. The air in the furnace was saturated

25 Upon removal from the furnace, the salt-coated discs exhibited substantial attack with localized penetration typically as shown in Fig. 1. The unsalted disc, however, proved to be quite resistant to this accelerated oxidation test and exhibited only a thin oxide scale and no localized penetration or pitting. 30

The same test performed on other alloys such as IN-738 and IN-792 did not develop pits, which is consistent with burner rig experience and supports the conclusion that this accelerated test is effective for inducing localized corrosion of susceptible alloys and, consequently, for distinguishing the susceptible from the non-susceptible materials. On that basis, corrosion disc test specimens of the alloys of Table I were prepared as described above for the same test. 40

TABLE I

| Alloy Designation | Co | Cr | Al | Ti | Mo | W | Ta | B | Zr | C | Ni |
|-------------------|-----|----|----|----|-----|---|-----|------|-----|-----|------|
| A | 9.5 | 14 | 3 | 5 | 4 | 4 | 0.5 | .016 | .03 | .15 | Bal. |
| B | " | " | " | " | 3.5 | " | 1.0 | " | " | " | " |
| C | " | " | " | " | 3 | " | 1.5 | " | " | " | " |
| D | " | " | " | " | 2.5 | " | 2.0 | " | " | " | " |
| F | " | " | " | " | 1.5 | " | 3.0 | " | " | " | " |

NOTE: Alloys A-D outside the invention
Alloy F inside the invention

45 Distribution of molybdenum, tungsten, tantalum and titanium in the carbide phase of René 80 and each of the Table I alloys is

set forth in Table II together with the results of the burner rig and accelerated oxidation tests. 50

TABLE II
Carbide (Wgt %)

| | Ti | Ta | W | Mo | (Mo + W) | Pitting Attack |
|---------|----|----|----|----|----------|----------------|
| René 80 | 51 | — | 18 | 18 | 36 | Yes |
| A | 43 | 13 | 19 | 11 | 30 | Yes |
| B | 36 | 24 | 18 | 8 | 26 | Yes |
| C | 36 | 31 | 12 | 6 | 18 | Yes |
| D | 32 | 40 | 11 | 4 | 15 | Possible |
| F | 27 | 48 | 12 | 2 | 14 | No |

55 On the basis of these results, further tests were made for the purpose of optimizing the composition in terms of the ultimately desired combination of mechanical and corrosion-resistance properties. Thus, a

total of 8 additional alloys constituting modifications of alloy F were prepared generally as described above, being cast as one-inch by four-inch by five-inch slabs at 1900°F mold temperature and 225°F metal 60

superheat. The nominal composition of each of these alloys is set out in Table III.

TABLE III

| Alloy | Ni | Co | Cr | Al | Ti | Mo | W | Ta | Cb | Hf | B | Zr | C |
|-------|------|-----|----|-----|----|------|---|-----|-----|-----|-------|-----|-----|
| G | Bal. | 9.5 | 14 | 3 | 5 | 1 | 4 | 2.5 | -- | -- | 0.015 | .03 | .12 |
| H | Bal. | 9.5 | 14 | 3.2 | 5 | 1.25 | 4 | 3 | -- | -- | | | |
| I | Bal. | 9.5 | 14 | 3 | 5 | 1.5 | 4 | -- | 1.5 | -- | | | |
| J | Bal. | 9.5 | 14 | 3 | 5 | 1 | 4 | 2 | 0.5 | -- | | | |
| K | Bal. | 9.5 | 14 | 3 | 5 | 1 | 4 | 0.5 | 1.5 | -- | | | |
| L | Bal. | 9.5 | 14 | 3 | 5 | 1 | 4 | 2 | -- | 0.5 | | | |
| M | Bal. | 9.5 | 14 | 3 | 5 | 1.5 | 4 | -- | -- | 2.5 | | | |
| N | Bal. | 9.5 | 14 | 3 | 5 | 1.5 | 4 | 3 | -- | -- | | | .15 |

5 Most of these slabs were heat treated as described above except that the first step involved heating at 2150°F., rather than 2225°F., as noted in Table IV.

10 The slabs were then sectioned and evaluated metallographically and standard tensile, rupture and corrosion disc specimens were machined and tested.

15 The tensile properties of all the above alloys were quite good. Thus, room temperature and 1200°F. test results were similar to what might be expected for thick

sections of standard René 80 superalloy, there being no significant difference in tensile strength or yield strength between the two sets of alloys. Ductility of the alloys was, in general, slightly improved with the 2150°F. solution heat treatment.

The results of preliminary rupture testing of these alloys are set out in Table IV and are shown graphically on the Larson-Miller charts of Figs. 8, 9 and 10, René 80 curves again being used for comparison.

TABLE IV

| Alloy* | Temp. (°F) | Stress (ksi) | Life (hrs) | P _{L-M} | Elongation Percent | RA Percent |
|--------|------------|--------------|------------|------------------|--------------------|------------|
| G | 1600 | 40 | 215.5 | (46.0) | 3.3 | 6.4 |
| H | " | " | 556.7 | (46.8) | 4.3 | 9.0 |
| J | " | " | 438.3 | (46.05) | 6.9 | 8.9 |
| K | " | " | 340.6 | (46.4) | 5.1 | 9.9 |
| L | " | " | 286.3 | (46.25) | 5.9 | 13.7 |
| M | " | " | 434.4 | (46.65) | 8.1 | 9.8 |
| G | 1400 | 90 | 37.7 | (40.4) | 6.2 | 10.0 |
| M | " | " | 57.2 | (40.5) | 9.3 | 27.0 |
| N-1 | 1800 | 27.5 | 32.2 | (48.6) | 6.8 | 15.0 |
| N-2 | " | " | 33.0 | (48.7) | 3.1 | 6.4 |
| N-1 | 1600 | 40 | 594.6 | (46.9) | 4.9 | 12.0 |
| N-2 | " | " | 1280.1 | (47.6) | 4.8 | 7.2 |
| N-1 | 1600 | 35 | 1509.2 | (47.7) | 3.8 | 6.4 |
| N-2 | " | " | 1862.1 | (47.9) | 5.8 | 7.0 |
| N-1 | 1400 | 90 | 27.7 | (39.9) | 4.2 | 19.0 |
| N-2 | " | " | 83.0 | (40.8) | 5.7 | 12.0 |

*N-1 series differ from the other series only in that the solution heat treatment step temperature was 2225°F. instead of 2150°F.

Figures 8 to 10 show plots of stress-rupture properties against Larson-Miller parameters (P_{L-M}) for alloys N-1 and N-2, alloys G and H, and alloys J, K, L and M respectively, using the figures given in Table IV above. As illustrated by Figs. 8-10, the properties of the alloys of this invention are generally excellent at 1600°F. and 1800°F., most of the test points falling around the average for René 80 and one point (Figure 8, $P_{L-M} = 46.9$ for alloy N-1 at 1600°F./40 ksi) lying one full parameter above the René 80 average. Rupture lives at 1400°F./90 ksi fall well below the René 80 average except for one case (alloy N-2, 2150° solution temperature) of coincidence. Rupture ductilities are generally lower than those of René 80 but not so low as to be of serious concern by themselves.

Corrosion tests carried out on these specimens were of three types. Accelerated oxidation tests as described above were run up to 1700 hours with no signs of pitting attack despite the presence of many carbides near the sample surface.

Electrochemical screening tests likewise yielded encouraging results, corrosion rates being approximately equivalent to that of alloy IN-738. Thus, for example, predicted penetration values (mils per 600 hours exposure at 1600°F) were 4.4 mils for alloy N, as compared to 3.3 mils for IN-738 and 7.7 mils for René 80.

Burner rig tests carried out at 1600°F on test specimens of most of the experimental alloys (including René 80) yielded results set forth in Table V.

TABLE V

| Alloy | Temp. (°F) | Time (Hrs) | Penetration (Mils) | |
|---------|------------|------------|--------------------|-----------|
| | | | Maximum | Average |
| G | 1600 | 651 | 1.35 | 0.45 |
| H | " | 614 | 3.8 | 1.4 |
| J | " | 629 | 9.95 | 5.1 |
| K | " | 603 | 1.3 | 0.6 |
| L | " | 651 | 1.75 | 0.6 |
| M | " | 651 | 2.2 | 1.2 |
| N-1 | " | 608 | 6.15 | 2.9 |
| N-2 | " | 1008 | 5.4 | 2.1 |
| René 80 | " | 611 | 4.0 | 0.65 |
| " | " | 650 | 11.7/3.65 | 7.50/0.57 |
| " | " | 1092 | 7.40/1.60 | 1.20/0.50 |
| " | " | 1017 | 5.7 | 1.7 |
| N-7 | 1800 | 1012 | 6.8 | 4.4 |
| René 80 | " | 507 | 17.8/31.9 | 3.1 |
| " | " | 884 | 17.97 | 9.53 |
| " | " | 458 | 18.18/31.9 | 8.73/31.9 |

The tests were conducted in a manner such that the specimens were exposed to an atmosphere generated from the combustion of a Number 2 diesel oil containing one percent sulfur to which was added 125 parts per million of sodium in the form of synthetic sea salt. The fuel was combusted in the air to fuel ratios normally found in a gas turbine, and the resultant combustion product was flowed past the specimens at 70 feet per second at one atmosphere pressure. The specimens (discs of one-inch diameter and 60 mils thickness) were maintained at the combustion gas temperature throughout the test period.

The sample specimens subjected to these burner rig tests were sectioned, mounted and examined metallographically to determine the depth of penetration of the

hot corrosion attack. The occurrence of a second number at some points in the table indicates that the two sides of the René 80 specimen exhibited very different rates of attack as, for example, where one side contains a localized pit and the attack has proceeded all the way through the specimen, while the other shows only normal surface attack.

The fact that pits have not been detected in any of the experimental alloys, during these tests is considered to be very important. In addition to the advantages thus to be gained as set out above, this will enable much more accurate prediction of the corrosion rate in service.

With regard to castability, we have found upon examination of as-cast slabs of alloys of this invention that there is no serious

problem of casting soundness. Since no attempt has been made to optimize casting parameters for these particular alloys, it is conceivable that any castability problems encountered in the pouring of actual parts can be overcome through development of optimum pouring parameters.

As indicated previously, we have discovered that the heat-treatment atmosphere is not critical in terms of the resistance of these new alloys to hot corrosion. Thus, they differ from related prior art alloys both in that they are highly resistant to pitting attack and are not rendered subject to rapid hot-corrosion attack by heat treatment in vacuum. Accordingly, in carrying out the present invention, the heat-treatment operation does not require use of a hydrogen or an argon atmosphere or necessitate a preliminary coating step for the protection of the workpiece.

Summarizing, this invention contemplates the use of a small but critical amount of tantalum, columbium, or hafnium or mixture thereof to eliminate the pitting attack on certain nickel-base superalloys of superior mechanical properties. The amount of tantalum or alternative additive should not be in excess of that which will produce significant microstructural instability (i.e., 3.5 percent) and should not be less than that necessary to produce the new results of this invention (i.e., 1.5 percent for mixtures and 1.0 percent for columbium, 2.0 percent for hafnium and 2.5 percent for tantalum used individually).

Whenever in this specification and the appended claims proportions, percentages or amounts are stated, reference is to the weight basis unless otherwise specifically indicated.

WHAT WE CLAIM IS:—

1. A nickel-base superalloy consisting of, by weight:

| | |
|----------------|----------------------|
| 13.7 to 14.3 % | chromium |
| 9.0 to 10.0 % | cobalt |
| 4.8 to 5.5 % | titanium |
| 2.8 to 3.2 % | aluminium |
| 3.7 to 4.3 % | tungsten |
| 1.0 to 1.5 % | molybdenum |
| 0.01 to 0.02 % | boron |
| 0.02 to 0.10 % | zirconium |
| 0.08 to 0.20 % | carbon and either |
| 1.5 to 3.5 % | of a mixture of |
| | tantalum, columbium |
| | and hafnium, or |
| 2.5 to 3.0 % | tantalum, or |
| 1.0 to 1.5 % | columbium, or |
| 2.0 to 2.5 % | hafnium; the balance |
| | being nickel, |

said superalloy consisting of a matrix, an α' precipitate and a monocarbide phase, distributed through the matrix, consisting of titanium, molybdenum, tungsten and/or tantalum and/or columbium and/or hafnium in proportions such that the total of molybdenum and tungsten is less than 15 percent of the carbide phase.

2. A superalloy as claimed in claim 1, wherein the amount of carbon is from 0.08 to 0.12% by weight.

3. A superalloy as claimed in Claim 1 consisting of, by weight:

| | | |
|------------|---------|---------|
| Chromium | 14 | percent |
| Cobalt | 9.5 | " |
| Aluminium | 3.0 | " |
| Titanium | 5.0 | " |
| Tungsten | 4.0 | " |
| Molybdenum | 1.5 | " |
| Tantalum | 3.0 | " |
| Boron | 0.015 | " |
| Carbon | 0.12 | " |
| Zirconium | 0.03 | " |
| Nickel | Balance | |

4. A superalloy as claimed in Claim 1 having the following composition:

| | | |
|------------|---------|---------|
| Chromium | 14 | percent |
| Cobalt | 9.5 | " |
| Aluminium | 3.0 | " |
| Titanium | 5.0 | " |
| Molybdenum | 1.0 | " |
| Tungsten | 4.0 | " |
| Tantalum | 0.5 | " |
| Columbium | 1.5 | " |
| Boron | 0.015 | " |
| Carbon | 0.12 | " |
| Zirconium | 0.03 | " |
| Nickel | Balance | |

5. A superalloy as claimed in Claim 1 having the following composition:

| | | |
|------------|---------|---------|
| Chromium | 14 | percent |
| Cobalt | 9.5 | " |
| Aluminium | 3.0 | " |
| Titanium | 5.0 | " |
| Molybdenum | 1.0 | " |
| Tungsten | 4.0 | " |
| Tantalum | 2.0 | " |
| Hafnium | 0.5 | " |
| Boron | 0.015 | " |
| Zirconium | 0.03 | " |
| Carbon | 0.12 | " |
| Nickel | Balance | |

6. A method of producing an article of a nickel-base superalloy, which method comprises preparing an ingot or a superalloy as claimed in any one of the preceding claims, remelting the ingot and casting the molten alloy to form the article,

and heat treating the thus-formed article in a vacuum or a neutral atmosphere.

7. A superalloy article when produced by the method of Claim 6.

5 8. A superalloy as claimed in Claim 1 sub-

stantially as hereinbefore described in any one of the Examples F to N, N1, N2 and N7.

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Fig. 1.

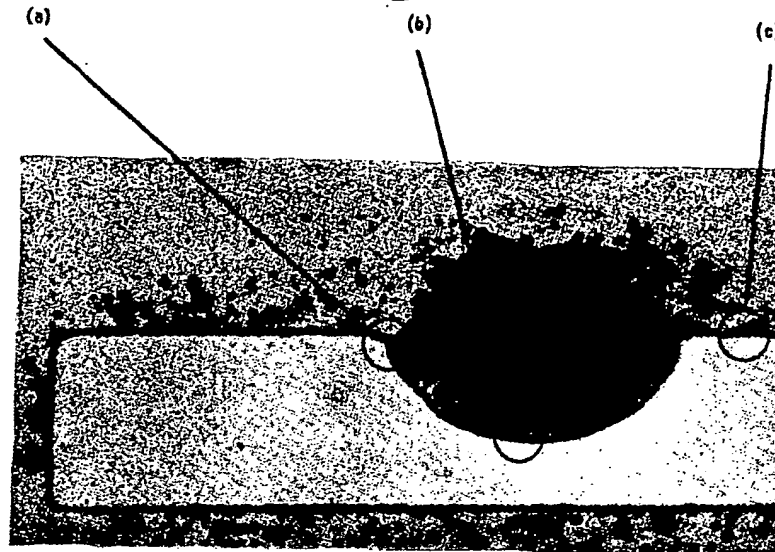
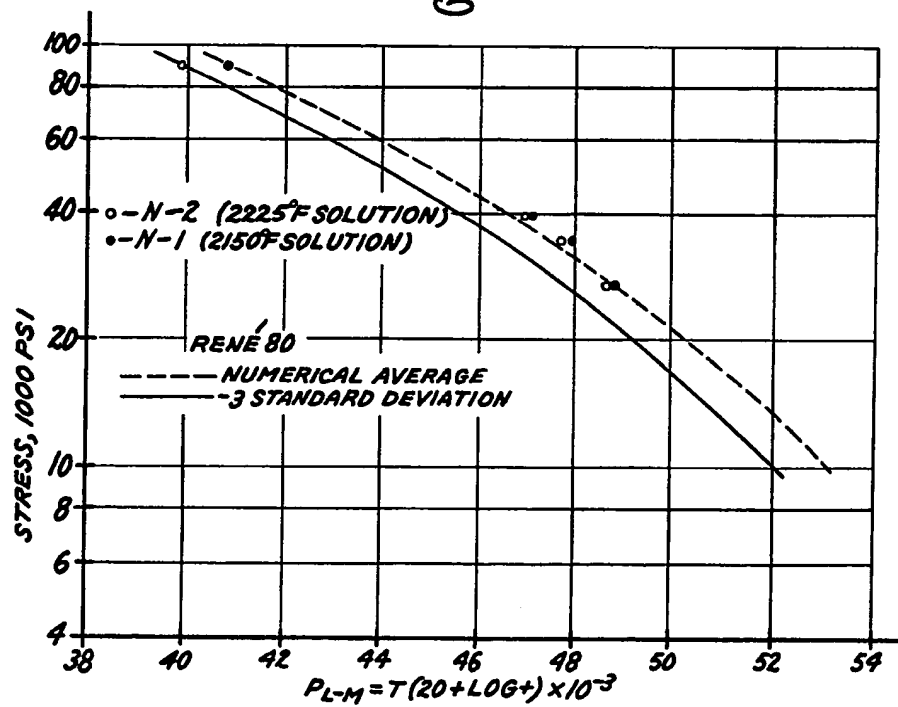


Fig. 8.



1 511 562

COMPLETE SPECIFICATION

5 SHEETS

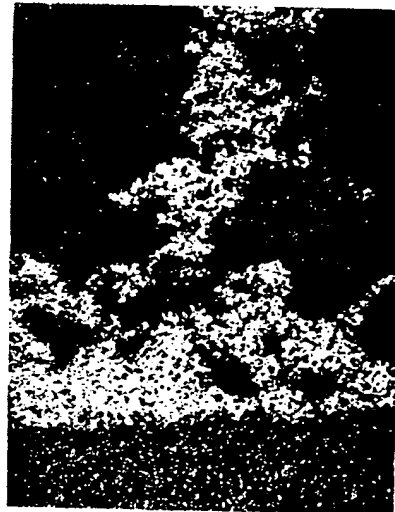
*This drawing is a reproduction of
the Original on a reduced scale.*

SHEET 2

Fig. 2.



Fig. 3.



J 511 562
5 SHEETS

COMPLETE SPECIFICATION

*This drawing is a reproduction of
the Original on a reduced scale.*
SHEET 3

Fig. 4.



Fig. 5.



1 511 562

5 SHEETS

COMPLETE SPECIFICATION

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SHEET 4

Fig. 6.

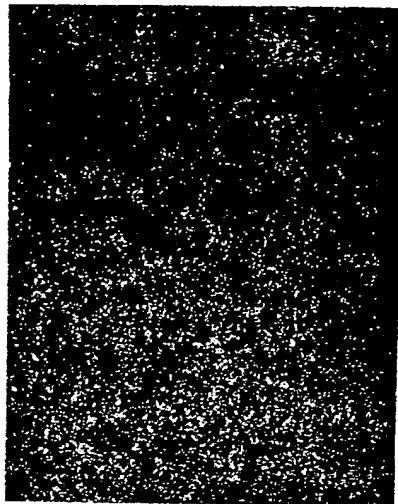


Fig. 7.

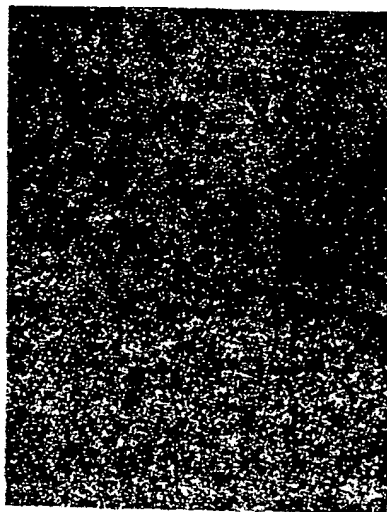


Fig. 9.

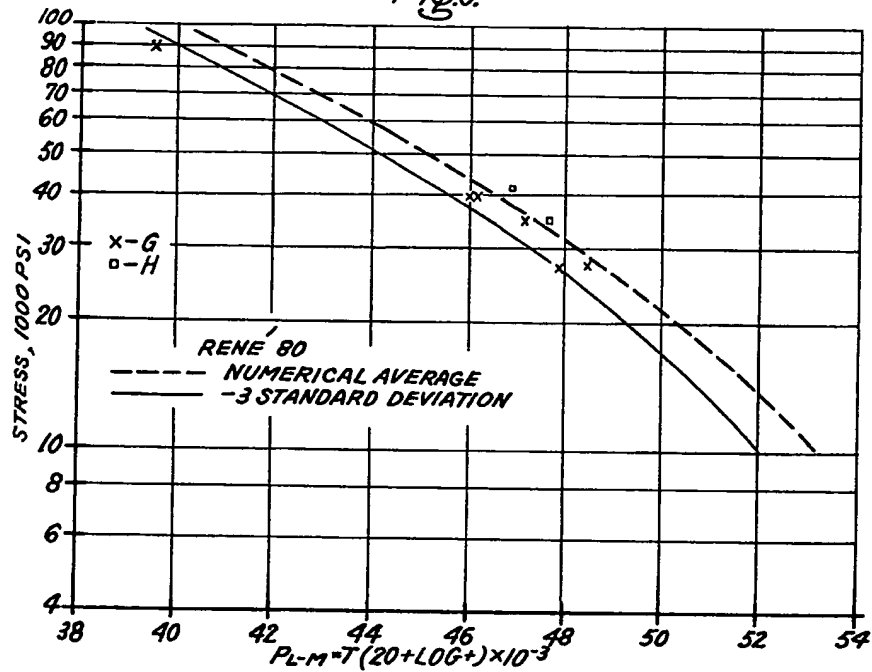


Fig. 10.

